Evaluation of Standards for a 10 V MAP Service at NIST

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Abstract

A Measurement Assurance Program (MAP) provides a method of assessing and maintaining the quality of a measurement process. Such a program has two parts: the operation of a set of statistical tools to ensure the predictable behavior of the standards and measurement systems in a laboratory; and a calibration of the system relative to the SI units from time to time to characterize its drift with time and eliminate its offsets. The need for the latter is best fulfilled through a closed-loop transfer designed to take advantage of the same statistical tools to analyze the uncertainty of the transfer. NIST MAP services are examples of such closed-loop transfers.

This paper describes the development of a 10 V MAP service and the problems associated with Zener standards[2] that are used as the transfer standards. The uncertainty of a MAP is determined by the behavior of the traveling standards and the measurement systems at NIST and the customer's lab. We will report the recent results of the temperature, pressure, and humidity characterizations for our pool of Zeners. Compensating for these effects will reduce the uncertainties contributed by the traveling standards. The calculation of uncertainty for a MAP transfer is also described.

Introduction

The most demanding instrument specifications for dc voltage measurements are seen at the 10 V level. Accordingly, the demand from industrial laboratories for lower uncertainties of 10 V calibration is increasing. Although an in-house Josephson voltage standard (JVS) can produce calibrations with the lowest uncertainties, it still requires levels of expertise and expense that render it impractical for widespread use. The majority of standard laboratories are still using Zener references or standard cells as their primary voltage standards. Many laboratories have shifted to the use of Zener standards for calibrations at the 10 V level. This has led to improved workload throughput, improved support for calibration of precision calibrators and meters, and to avoidance of shipping problems that are inherent with standard cells. NIST has been offering a MAP service at 1.018 V for many years. A comparable MAP service is needed to support 10V

[1] Electricity Division, Electronics and Electrical Engineering Laboratory, Technology Administration, U.S. Department of Commerce. Official contribution of NIST; not subject to copyright in the United States.

[2] A Zener voltage standard is a commonly used solid-state electronic voltage standard based on Zener diode maintained in a stable temperature oven and provided with supply batteries for mains-independent use.

calibrations. We are now characterizing commercially available Zener standards at the 10 V level for their noise, pressure, temperature, and humidity properties. A group of these characterized Zener standards will be used as the transfer standards in a NIST MAP service at 10 V and to improve the uncertainties of NIST calibrations.

The output values of Zener standards are known to be affected by environmental changes such as temperature, barometric pressure, and relative humidity [1,2]. Some models of commercial Zener standards can also exhibit seasonal changes in their 1.018 V and 10 V outputs [3]. In the Zener MAP service, a set of Zener standards is measured at NIST over a few weeks time frame and then sent to a customer laboratory. There they are measured using the customer's measurement system in the way it is normally used for calibrations. After a specified number of measurements have been made, the Zener standards are then returned to NIST for further measurements. The traceability of the customer's standards to the U.S. representation of the SI volt is then derived from an analysis of the NIST and customer data. Environmental conditions at NIST and the customer's laboratory can be different. Lack of knowledge of the travelling Zener standards' responses to varying environmental conditions can result in errors in the results of the transfer as well as mis-estimation of its overall uncertainty. Characterizing a set of transportable Zener standards for voltage variations due to environmental effects can provide an accurate statistical model, use of which will reduce the likelihood of error and achieve the best uncertainty for the MAP procedure.

Characterization of Zener standards

Three environmental conditions; temperature T, barometric pressure p, and relative humidity H, are known to influence a Zener's output. Since historical data support the use of a linear model to describe its drift with time, a Zener standard's output can be expressed by

$$U(t, p, R, H) = U_0 + c_t t + c_p(p-p_0) + c_R(R-R_0) + h(H) + \varepsilon \quad (1)$$

where U_0 is the Zener value at an initial time, c_t is the drift rate of the Zener output with time t, c_p is the pressure coefficient, c_R is the temperature coefficient expressed by Zener thermistor resistance, p_0 and R_0 are the reference points of pressure and thermistor, h(H) is the correlation function for relative humidity, and ε is the intrinsic noise of the Zener standard. Once a Zener is characterized for c_p, c_R and h(H), Eq.(1) can be simplified as

$$U(t) = U_0 + c_t t + \varepsilon' \quad (2)$$

where ε' is the total noise that includes all sources, and $U(t) \equiv U(t, p, R, H) - c_P(p-p_0) - c_R(R-R_0) - h(H)$, which is the Zener reference output that is corrected for environmental conditions. The uncertainty of the MAP is now determined by the intrinsic noise of the Zener standards, the uncertainties in the NIST and customer measurement systems, errors in the correction factors, and transfer factors of the Zener standards.

At NIST, a pressure chamber and a temperature / humidity chamber were used to measure the pressure and temperature coefficients, and to characterize the humidity correlation function h(H). A 10 V Josephson voltage standard (JVS) system is dedicated to measuring the Zener outputs under variable environmental conditions to avoid drift in the measurement system and further complication of the data analysis.

Pressure coefficient c_p

The pressure chamber is a cylindrical vessel of 20 cm in diameter and made of Plexiglas. The chamber pressure is monitored by a digital barometer with an accuracy of ± 0.2 hPa and controlled by a mechanical pump with a gas handling system. The pressure stability inside the chamber during a Zener calibration can be maintained within ± 0.3 hPa. Figure 1 shows a typical calibration result of a Zener standard with pressure between 750 hPa and 1000 hPa. The measurements were made usually within 4 hours, so that the Zener drift in the measurement period was negligible. The pressure coefficients for 20 ZenerA[3] and 8 ZenerB standards have been measured. No noticeable hysteresis effect on a Zener output due to pressure changes was observed. ZenerA standards manufactured in recent years usually have pressure coefficients in the range between 10 nV/hPa and 20 nV/hPa with most being around 20 nV/hPa. Earlier ZenerA standards using different components have pressure coefficients around –1 nV/hPa. The eight ZenerB standards that were measured had pressure coefficients mostly in the vicinity of 5 nV/hPa and two had pressure coefficients around 1 nV/hPa.

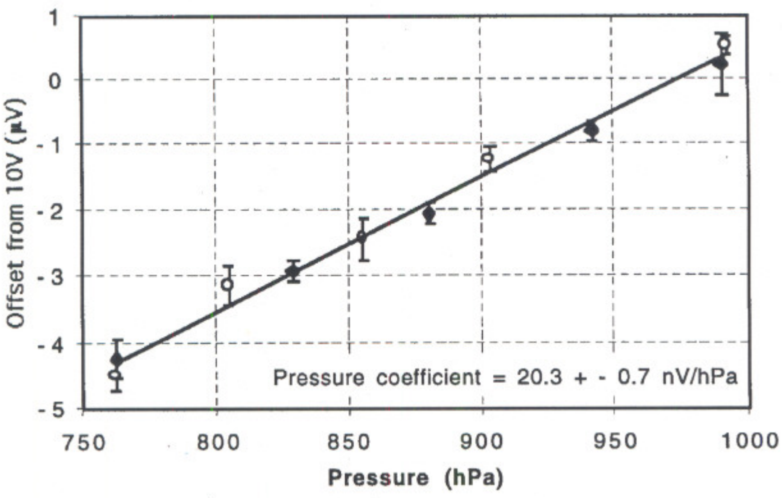

Fig. 1 A typical Zener output vs. pressure. The data points "♦" were taken with pressure ramping downward, and data points "○" with pressure ramping upward. The standard uncertainty of the pressure coefficient for this example is 0.7 nV/hPa.

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[3] Zener voltage standards from two manufactures were evaluated. The standards are designated ZenerA and ZenerB.

The relative standard uncertainty of the pressure coefficient measurements is normally a few percent of the coefficient. The pressure coefficient measurements' Type B uncertainty u_P is given by

$$u_p = u_{C_p}(P_{NIST1} - P_{Lab}) \qquad (3)$$

where u_{Cp} is the standard uncertainty of the pressure coefficient measurements and P_{NIST} and P_{Lab} are the mean pressures in the corresponding laboratories during the MAP process. The magnitude of this contribution can range from a few parts in 10^{-10} up to 1.5×10^{-8}, depending on the magnitude of the pressure coefficient and its measurement uncertainty.

Temperature coefficient c_R

In the following example, we describe a procedure to measure the temperature coefficient c_R of a Zener voltage standard, i.e., its change in output voltage per unit change in resistance of its internal monitoring thermistor. An environmental chamber is used to expose the standard to a range of ambient temperatures. Each change has the same effect as altering the set point temperature of the standard's internal oven slightly. The standard's voltage and thermistor resistance are monitored throughout the test and c_R calculated from the results. While c_R is being measured, the humidity inside the chamber is maintained at a constant level, e.g. 50 %. The chamber's temperature is then adjusted and allowed to reach a stable state. This takes about 5 minutes. During a week of measurements, the chamber temperatures were adjusted in a sequence of 22.5, 27.5, 22.5, 31.4, 19.5, 22.5, 16.5 and 22.5 °C. The temperature stability in the test chamber is ± 0.1 °C for this temperature range. The temperature 22.5 °C was used as the base temperature to monitor the Zener drift during the measurement interval of a week. While the chamber temperature was being changed, the Zener voltages were continuously measured against the JVS and the thermistor changes were recorded by a DVM. The time it took for a thermistor to reach a stable state after a temperature change was approximately four hours, and the Zener was measured for at least 6 hours after attaining a stable state.

In the first order, the thermistor resistance tends to vary linearly with the temperatures. The thermistor changes of the four ZenerA standards were seen to be around -50 Ω/°C. The measurements of the Zener output at 22.5 °C at various times established a base line for the offset measurements at other temperatures. A mean difference between base line temperature measurements and the measurements at another temperature can be calculated. The temperature coefficient, c_R, is determined from the mean differences and thermistor readings as shown in Figure 2. The temperature coefficients of four ZenerA standards ranged from approximately 1.6 nV/Ω to 5.4 nV/Ω. The uncertainty of the coefficients ranged from 0.1 nV/Ω to 0.8 nV/Ω, with the main contribution being the noise level of the Zeners. Similarly, the temperature coefficient measurements' Type B uncertainty contribution u_R is given by

$$u_R = u_{C_R}(R_{NIST} - R_{Lab}) \qquad (4)$$

where u_{CR} is the standard uncertainty of the temperature coefficient measurements and R_{NIST} and R_{Lab} are the mean resistances of thermistor in the two laboratories during the MAP process. The temperatures in most metrology labs are controlled in a range between 22 °C and 24 °C, and the difference between the mean thermistor resistances in the two laboratories is typically within 100 Ω for ZenerA used as the travelling standards. The uncertainty of the temperature coefficient is typically about 1×10^{-8}.

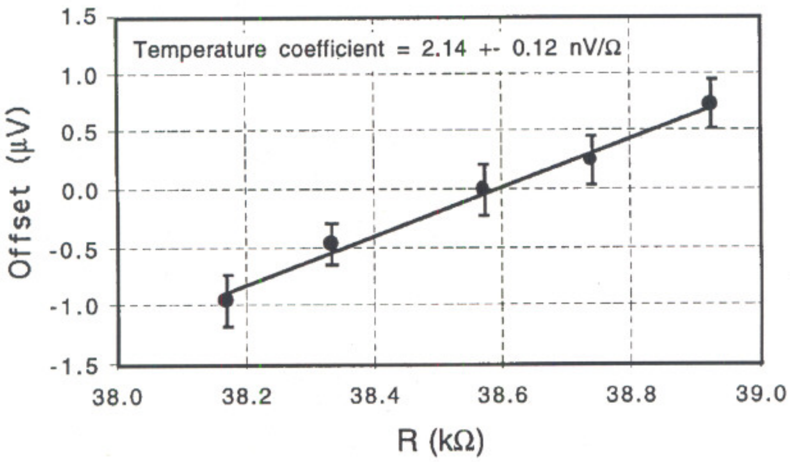

Fig. 2 A linear LSS fit of thermistor reading vs. Zener offset using a reference point corresponding to the environmental temperature of 22.5 °C. The error bars are the standard deviations of data obtained from the offset measurements at specific temperatures. The standard uncertainty of the temperature coefficient is 0.12 nV/Ω for this measurement.

Humidity correlation function, *h(H)*

The output changes with time of a Zener standard as a function of humidity are complicated owing to the time constants involved. If such changes were significant, the linear models used to predict transfer results would be inadequate and much more sophisticated measurements and models would have to be used for predictions or the accuracy of the transfer would be limited. Therefore, a suitable Zener standard would have a negligible response to humidity changes. Four ZenerA and four ZenerB standards were tested for their humidity response using an environmental chamber. The chamber's relative humidity can be regulated from 10 % up to 98 % with a stability of ± 2.5 % and can reach a stable condition within 20 minutes after a new setting. The eight Zener standards were kept in the chamber for three months. The humidity of the chamber was set in a sequence of 50, 25, 70, 25, and 50%. Continuous measurements were taken for three weeks at each relative humidity setting. Figures 3 and 4 show the response of a ZenerB standard and ZenerA standard to relative humidity, respectively. The small number of Zeners that we tested does not have adequate statistical significance to allow inference of the general behavior of each type of Zener standard. Only for the particular Zener standards that we have tested, we found the ZenerAs do not show significant correlation between the voltage outputs and relative humidity while the ZenerB exhibit strong correlation between the drift rate of the voltage outputs and relative humidity. The time constant for any of the tested ZenerBs to reach new stabilized drifting rates is only a few days. A change of Zener drift rate due to humidity can complicate the MAP process and can impose an uncertainty factor on the data analysis. If possible, it is beneficial to choose the travelling standard without significant correlation between its output and relative humidity.

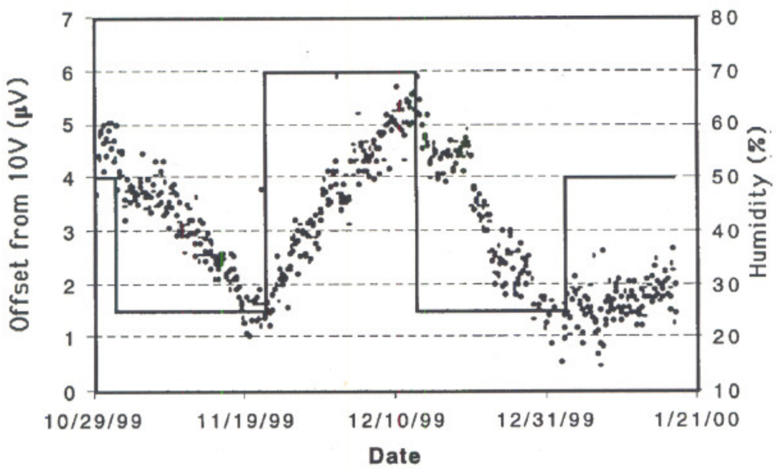

Fig. 3 Effect of relative humidity on a single typical ZenerB standard. The steps are the relative humidity of the environmental chamber. The data points are the voltage outputs from the Zener standard.

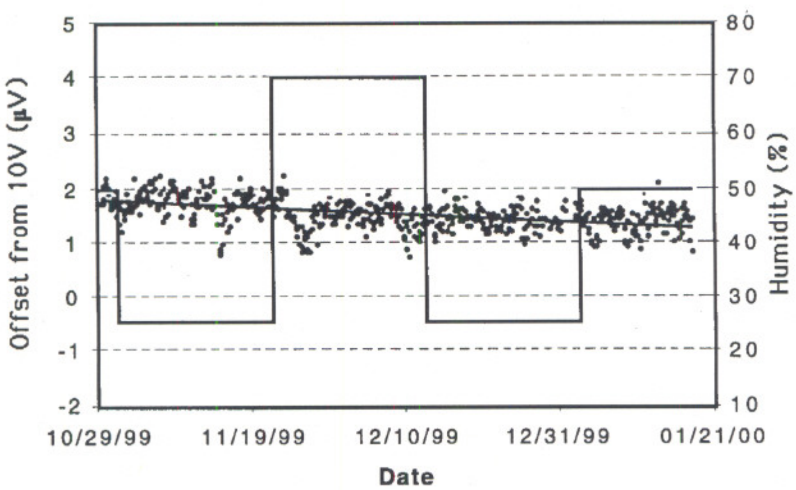

Fig. 4 Effect of relative humidity on a single typical ZenerA standard. The steps are the relative humidity of the environmental chamber. The data points are the voltage outputs from the Zener standard.

Uncertainty

The uncertainty of a MAP consists of contributions from Type A uncertainties of the measurement process in NIST and a customer's laboratory, variability of the travelling Zener standards, and Type B uncertainty components arising from the measurement systems and other sources. Let us assume we have a MAP that uses M travelling Zener standards. Each Zener standard is measured N times in the customer's laboratory and at NIST (N/2 times measurements before and N/2 times after the customer laboratory's measurements)[4]. In practice, M often is equal to four or three. The difference between the measurements at NIST and a customer's laboratory can be calculated by Eq.(5)

$$\Delta V = \frac{1}{MN}\sum_{i=1}^{M}\sum_{j=1}^{N}(V_{ij}(Lab) - V_{ij}(pred.)) \qquad (5)$$

where $V_{ij}(Lab)$ is the jth measurement for the ith Zener standard in the customer's lab after the corrections due to pressure and temperature effects, $V_{ij}(pred.)$ is the predicted value for customer's jth measurement for the ith Zener standard based on the linear regression using NIST before and after data. Figure 5 shows typical measurement results for a travelling Zener standard in two laboratories.

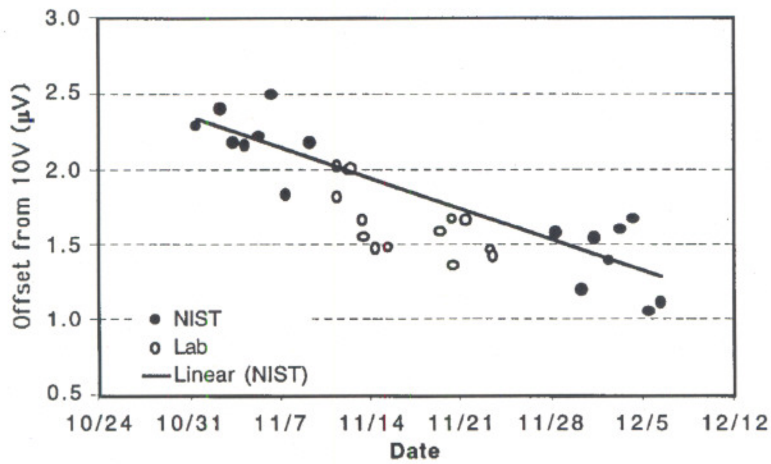

Fig.5 A MAP example. NIST takes 8 measurements first, a customer lab takes 16 measurements, and NIST takes another 8 measurements. The line is a LSS fit to NIST data only. The difference between the measurements at NIST and the customer laboratory can be found by the Eq.(5). The protocol can be applied to any two laboratories for an intercomparison.

The pooled standard deviation or the Type A uncertainty from the NIST measurements is obtained from Eq.(6)

[4] In general, the number of measurements at NIST can be different from that in the customer's laboratory.

$$u_{A,NIST}^2 = \frac{1}{M}\sum_{i=1}^{M} u_{i,NIST}^2 \quad (6)$$

where $u_{i,NIST}$ is the standard deviation of the mean of the linear regression fit for the ith travelling Zener standard at NIST. Similarly, the pooled standard deviation or Type A uncertainty of the customer laboratory's measurements is calculated by Eq.(7)

$$u_{A,Lab}^2 = \frac{1}{M}\sum_{i=1}^{M} u_{i,Lab}^2 \quad (7)$$

where $u_{i,lab}$ is the standard deviation of the mean for the linear regression fit for the ith travelling Zener standard at the customer laboratory.

The variability resulting from the transportation effect is evaluated by the Type B standard uncertainty of the mean difference between NIST and customer laboratory for all M travelling Zener standards by Eq.(8)

$$u_{B,transfer}^2 = \frac{1}{M(M-1)}\sum_{i=1}^{M}(\Delta V_i - \frac{1}{M}\sum_{i=1}^{M}\Delta V_i)^2 \quad (8)$$

where $\Delta V_i = \frac{1}{N}\sum_{j=1}^{N}(V_{ij}(Lab) - V_{ij}(pred.))$ represents the mean difference of the ith Zener standard at NIST and customer's laboratory.

The Type B uncertainty of a MAP includes uncertainties of the NIST measurement system $u_{B,NIST}$, and the customer measurement system $u_{B,Lab}$, and uncertainties of the pressure and temperature coefficient measurements expressed in Eq.(3) and Eq.(4).

$$u_B^2 = u_{B,NIST}^2 + u_{B,Lab}^2 + u_{c_p}^2 + u_{c_R}^2 \quad (9)$$

The combined standard uncertainty u_c can be obtained by the following Eq.(10).

$$u_c^2 = u_{A,NIST}^2 + u_{A,Lab}^2 + u_{B,transfer}^2 + u_B^2 \quad (10)$$

The transfer effect and measurements at NIST and customer's laboratory are not uncorrelated. A prudent estimation of the combined variance is used by summing up the variances of all sources due to the lack of detailed information of the correlation between the random noise of each Zener standard and the variability among the Zener standards.

The following table lists the uncertainty components and the associated degrees of freedom (DOF).

Source	Uncertainty	DOF
Pooled Type A uncertainty of NIST,	$u_{A,NIST}$	N-2
Pooled Type A uncertainty of customer	$u_{A,Lab}$	N-1
Type B uncertainty of Zener variability	$U_{B,transfer}$	M-1
Type B from NIST, customer systems, pressure, and temperature	U_B	∞

In order to estimate an expanded uncertainty with a certain confidence level, the effective degrees of freedom, $\nu_{eff,}$ can be calculated by the Welch-Satterthwaite formula according to the *Guide to the Expression of Uncertainty in Measurement (GUM)* [4]

$$\nu_{eff} = \frac{u_c^4}{\frac{u_{A,NIST}^4}{N-2} + \frac{u_{A,Lab}^4}{N-1} + \frac{u_{B,transfer}^4}{M-1}} \qquad (11)$$

The Student *t* factor corresponding to a certain confidence level for the effective degrees of freedom, $\nu_{eff,}$ can be found from the Table G.2 of *Guide to the Expression of Uncertainty in Measurement (GUM)* [4]. Consequently, the expanded uncertainty of the MAP for the assigned level of confidence is tu_c.

Conclusion

Pressure and temperature coefficients of a pool of Zener standards have been determined. Adjusting the output of the travelling Zener standard for a pressure change caused by a geological location and local weather conditions, can eliminate or minimize the Zener pressure effect in a MAP. Similarly, the temperature effect on the travelling Zener standard due to different laboratory conditions can also be eliminated. The uncertainties from the pressure and temperature coefficient measurements should be contributions to the total uncertainty budget. Zener standards have different responses to relative humidity depending on the model, manufacturer, and components. The humidity effect of a travelling Zener standard should be characterized before it is used in a MAP. A Zener standard with a strong correlation to relative humidity will have an additional uncertainty that may make it less suitable for MAPs with a low uncertainty requirement. Among the dozen Zener standards that have been tested, only two Zeners have been chosen as travelling standards. The search for additional qualified Zener standards will continue until the MAP requirements are satisfied.

Acknowledgement

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